In Situ Geochronology as a Mission-Enabling Technology

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Abstract— Although there are excellent estimates of ages of terrains on Mars from crater counting, even a few absolute ages would serve to validate the calibration. Results with uncertainties, although much larger than those that could be achieved in labs on Earth, would be extremely valuable. While there are other possibilities for in situ geochronology instruments, we describe here two alternative technologies, being developed in JPL. There are two common features of both. The first is analysis by means of miniature mass spectrometer. The second is use of laser sampling to reduce or avoid sample handling, preparation and pre-treatment and equally importantly, to allow analysis of individual, texturally resolved minerals in coarse-grained rocks. This textural resolution will aid in selection of grains more or less enriched in the relevant elements and allow construction of isochrons for more precise dating. Either of these instruments could enable missions to Mars and other planetary bodies.

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1. INTRODUCTION

The need for in situ dating

Determination of absolute ages for Martian rocks, both igneous and sedimentary, would be of great value. At present the only method available for determining ages of Martian surface rocks is crater counting [1]. Despite advances in estimating uncertainties [2], it is not very precise, and in the absence of an absolute calibration, its accuracy is unverifiable because of uncertainties in cratering rates. An earlier analysis suggested that the uncertainty in the ages of the major epoch boundaries might be as much as 2 billion years [3]. Therefore, absolute dating of a few critical samples could calibrate the cratering rate and give essential information on the historical evolution of Mars. Sample return would solve the problem, but the return stage of the currently planned sample return mission is not yet scheduled. So, even a precision of 10% for an in situ date would be a very significant advance on current knowledge. Thus, a limited, dedicated geochronology mission would be a very attractive opportunity, but at present there are no instruments for dating of rocks.

Isotopic Basis of Geochronology

Dating requires analysis of parent and daughter isotopes and ideally a range of ratios of parent-to-daughter so that isochron ages can be calculated. We describe here two approaches, each of which offers different advantages in terms of dating but share a common sampling approach of using a laser to produce material for analysis from either a single small area or a selected area of a sample, for example a specific mineral. The first isotope system involves the decay of ^{40}K to ^{40}Ar. Although doubt has been cast on its applicability to Martian rocks because of the presence of excess ^{40}Ar [4], it may prove invaluable in dating sedimentary materials ands thus the timing of aridification on Mars [5]. The other approach will implement Rb/Sr dating, depending on the decay of ^{87}Rb to ^{87}Sr, which has been used extensively for Martian meteorites [6]. For Ar analysis a separate aliquot of the sample would be fused at 1500°C.

Current State of the Art

There have been two principal but different approaches to develop an in situ instrument for making isotopic measurements from which dates could be produced. A team led by Tim Swindle [7] exerted a considerable effort to develop a potassium-argon instrument called AGE (Argon Geochronology Experiment). The design involved measurement of potassium concentration by Laser-Induced Breakdown Spectroscopy (LIBS) and release of argon by fusion of the sample at 1500°C. To determine the Ar concentration, the volume of rock from which Ar was released was measured by estimating the volume of the melted sample and from its density calculated from its elemental composition. However, the development was suspended at least three years ago [8].

One of the problems in determining an Rb-Sr age is the need to separate the measurement of ^{87}Rb and ^{87}Sr. In terrestrial laboratories Rb and Sr are separated chemically before analysis. The approach developed by Scott Anderson and his co-workers elegantly circumvents the problem by use of

RIMS (Resonance Ionization Mass Spectrometry). Sampling is done by Laser Desorption (LD) and the combined approach is referred to as LDRIMS [9]. The sampling laser produces, as expected, relatively few ions but the plume of mainly neutral species subjected to a second laser, finely tuned to ionize only strontium and followed a few microseconds later by another laser, which selectively ionizes rubidium. Because of this exceptional ionization efficiency, the current prototype can measure the isotope ratio of lab standards with 10 ppm Sr or Rb to a precision of 0.1% (1σ) and has been used to produce a geochron age for a terrestrial rock [10].

Our Approaches

Because of the two very different requirements for measuring Ar gas in one case and low concentrations of Rb and Sr in the other, as described above, two analytical approaches are being developed. However they share the common initial sampling method, laser ablation and the use of the same miniature mass spectrometer. The main difference then is the method for ionization of the sample and the approach to measurement of two isotopes at the same m/z value. The miniature mass spectrometer (MMS) has a double sector mass analyzer with Mattauch-Herzog geometry as described before by Sinha et al. [11]. This device utilizes a focal plane modified-CCD based ion detector array, which has 2140 elements (20 microns by 2 millimeter) and thus captures the whole spectrum simultaneously. We describe here the two concepts, both of which are still in development although neither has progressed to the stage of being a full instrument.

2. POTASSIUM-ARGON DATING CONCEPT

Sampling and the miniature mass spectrometer

This instrument would use laser sampling to generate a plume of analyte from ~500 micron diameter areas of target materials. Although some ions are produced the majority of the yield consists of neutral species. The plume of material is ionized by electron impact. Initially we had some problems because of interference from ions generated from background gases in the chamber. This was solved by inserting a slit between two plates through which the beam passes. It is isolated from the rest of the system and at ground potential but by applying a potential bias of 70v the beam can be deflected and does not enter the mass spectrometer source. Allowing passage of ions through the slit only for a short time after the laser pulse eliminated the interference problem. The relationship of the sample and laser beam to the mass spectrometer is shown in Figure 1.

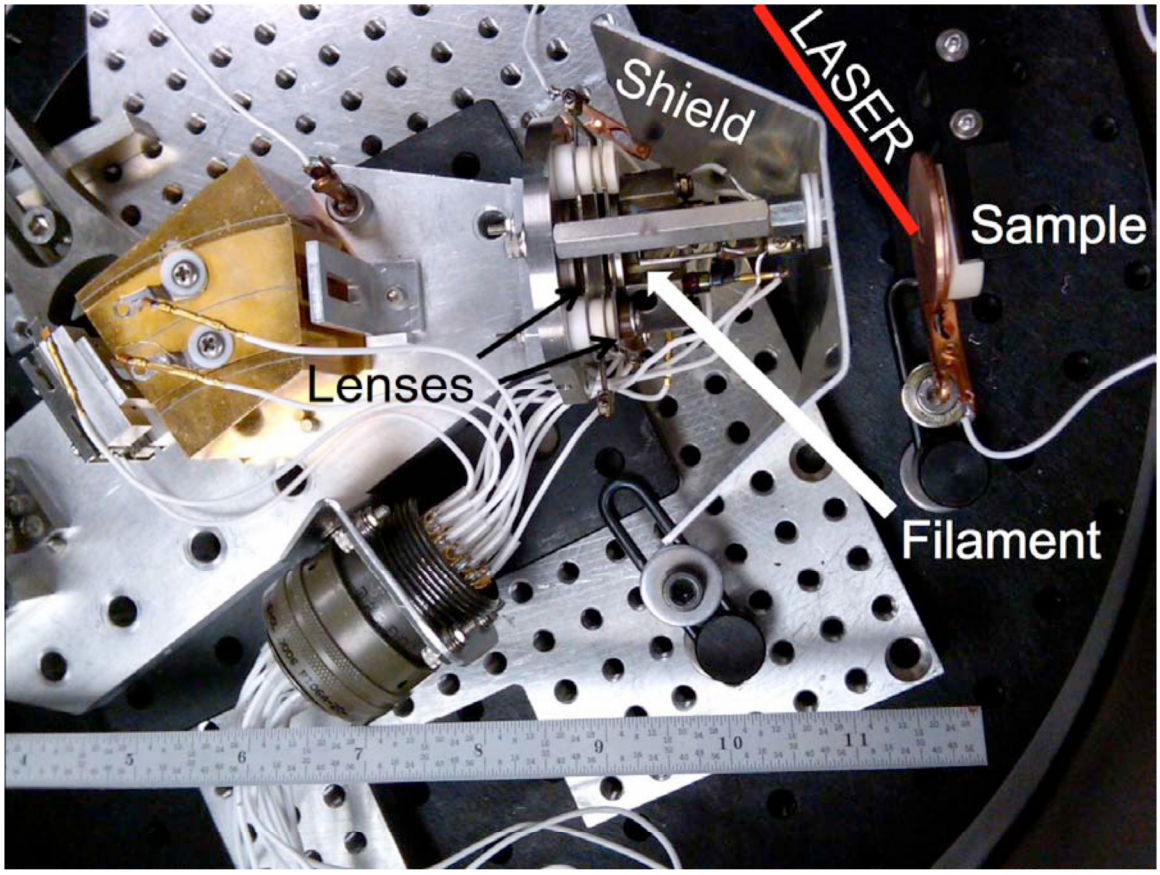

Figure 1. The relationship of the sample, laser beam and Miniature Mass Spectrometer (MMS). The laser path is oblique to the surface of the sample but the plume of material is ejected orthogonal to the surface towards the filament of the mass spectrometer. Both potassium and argon will be analyzed from the same volume of material.

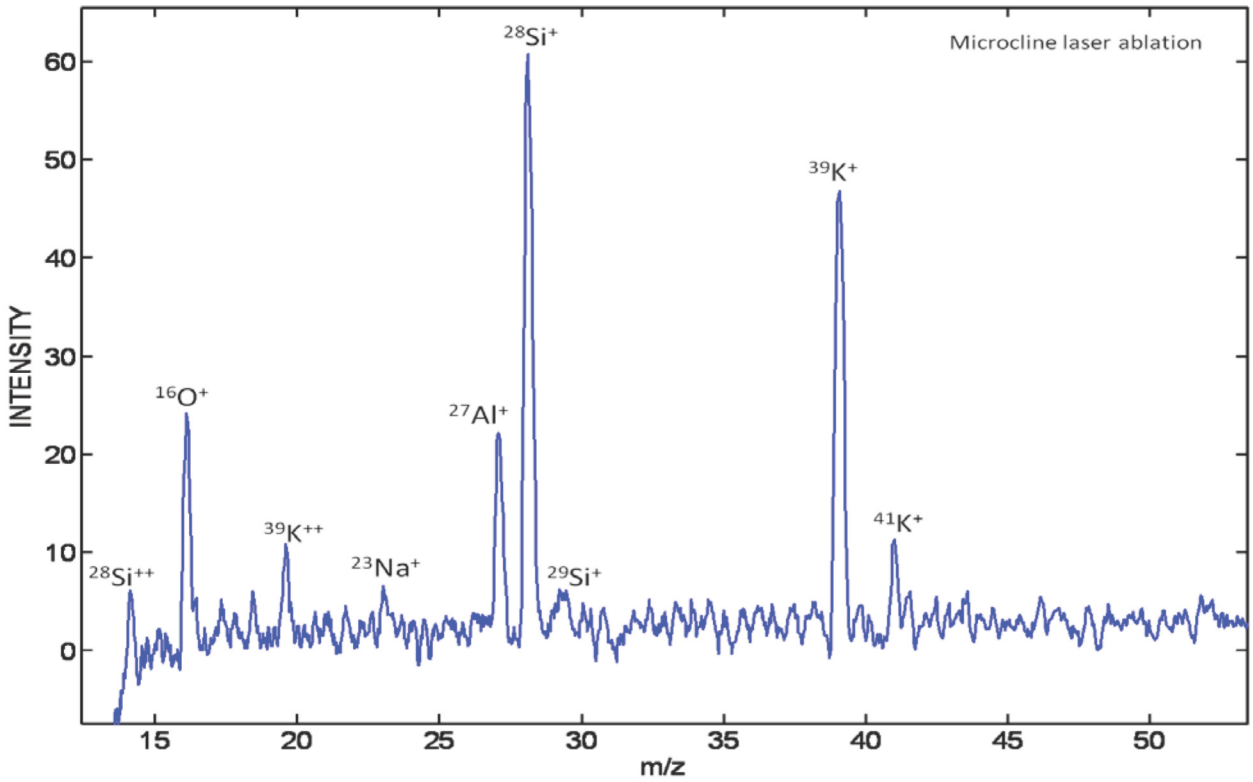

Figure 2. Mass spectrum from laser sampling of the potassium feldspar mineral, microcline. It shows clear resolution of the m/z 39 and 41 potassium isotopes [8] using a Nd-YAG laser at 1064 nm.

Initial Results

In our tests to date we have obtained good analyses of potassium with a Nd-YAG laser at fundamental frequency (1064 nm wavelength) run in Q-switched mode delivering 200 mJ pulses of 5 ns duration at a repetition rate of 5Hz [12]. We show a typical spectrum Figure 2. However, preliminary results from our more recent test using a frequency doubler to produce green laser light at 532 nm wavelength has given a greatly improved signal-to-noise ratio. In this case, the isotope of interest is ^{40}K, which occurs at an abundance of 0.0117 atom %. This does not pose a problem since all potassium that we can measure has the same abundance. Thus by measuring the ^{39}K and ^{41}K peaks 93.3 and 6.73 atom%, respectively, a correction can be made for any isotopic fractionation caused by the sampling or measurement. This avoids another possible problem of mass interference of ^{40}Ar with ^{40}K. However, it is still necessary to measure the ^{40}Ar.

Future Development

The initial results so far, although preliminary, are very encouraging, especially, the accuracy of the ratios of the various elements of interest, for example, silicon and aluminum and the values of the isotopic ratios. The next stage in the development would be to obtain a full argon isotope spectrum from a range of samples, for which potassium analyses have already been made. We will perform the analysis in a closed un-pumped chamber and accumulate all the Ar produced by the laser ablation sampling. The abundance of ^{40}Ar depends on the age of the sample, that is the amount of time during which it has accumulated by decay of ^{40}K since the last time the system was reset. Argon and other rare gases will be concentrated by removing water and other volatiles species produced by the laser shots using a getter pump before making a mass spectrometer isotopic analysis. It is important to note that the K and Ar come from the same volume of ablated sample. Therefore, after appropriate calibration of the mass spectrometer response to the different elements we will have a determined the $^{40}Ar/^{40}K$ ratio (and thus age). At that stage, it should be possible to analyze the uncertainties and their causes very well. It is expected that regardless of the precision achieved it is likely that use of calibration standards would be necessary to achieve the accuracy required. We have made a preliminary model to estimate the expected precision in age and it is of the order of ±10-20%, relative. This measurement will be suitable for determining the formation ages of K-poor materials (e.g., mafic igneous rocks) older than a few 100's of million years, and K-rich materials (e.g., clays, jarosite salts) older than a few 10's of million years. In both cases the uncertainty in the age would improve for older samples.

3. RUBIDIUM-STRONTIUM DATING CONCEPT

Analytical Approach

The second example is intended to produce Rb-Sr ages but also will offer rapid, multi-element as well as isotopic analysis capability with orders of magnitude improvements

in detection limits, dynamic range, and spatial resolution compared to the state-of-the-art for in situ instruments.

The standard terrestrial laboratory instrument for very high sensitivity analyses achieves its objectives by extremely efficient ionization of the sample. In the laser sampling version of the instrument the ablated material is transported in a flow of gas and introduced into a radio-frequency inductively-coupled Ar plasma. Part of the plasma is extracted through a differential pumping system and analyzed in a mass spectrometer. Ground-based instruments initially used laser ablation ionization coupled directly with mass spectrometers (LA-MS), as proposed for space applications [13, 14, 15]. LA-MS suffers from three problems, however. Firstly, the large ion velocity distribution makes focusing into a mass spectrometer difficult [16]. Second, the ion content of ablated material is not necessarily representative of the bulk material and ionization efficiency varies with different sample matrices, making quantitative analysis difficult [17]. Finally, in an ambient gas the laser ablation plume cools and condenses into an aerosol of fine, uncharged particles through interactions with the background gas [18]. These uncharged particles cannot be detected by mass spectrometers or transmitted using conventional or ion funnel [19] charged particle optics. LA-MS therefore requires sampling in vacuum, which limits sample size or requires extensive rock processing. Because of these shortcomings, LA-MS machines marketed in the 1970s and 1980s have now disappeared, and LA-Plasma Ionizer-MS instruments, like we propose here, are the tools of choice for spatially-resolved geochemical analysis on Earth [20, 21].

We have started development of a flight instrument equivalent of the inductively coupled plasma ionization coupled to multi-collector mass spectrometry (ICP-MC-MS). We have named the concept PLASMA (**P**lasma and **L**aser **A**blation **S**ystem for **M**ulti-element **A**nalysis but here will concentrate on its isotopic analysis capability. The schematic design of the instrument is shown in Figure 3.

Use of Mars Atmosphere as the Plasma Gas

For Mars deployment it would use ambient Martian atmosphere as the plasma gas, instead of the more usual argon. The Martian atmosphere consists of approximately 95% CO_2 [22].

It has one other key feature: it avoids sample handling problems by not having any sample handling capability. It is planned that it should sit on a rock surface directly with Mars ambient atmosphere in the sampling/plasma chamber and spots or areas for analysis selected for laser ablation. In the lab we have demonstrated stable operation of a plasma at power levels of 200 to 600 W with 6 torr of CO_2 to simulate Martian environment conditions. Furthermore, we have even produced an optical spectrum from a sample in the plasma.

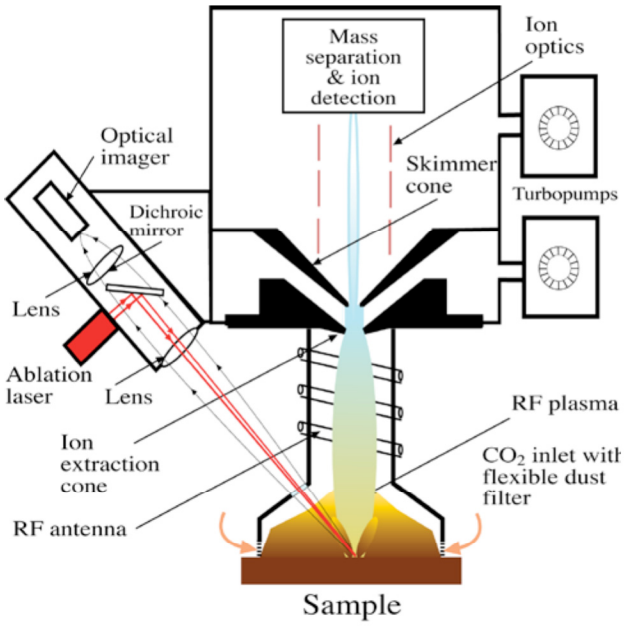

Figure 3. Conceptual design of the PLASMA Laser ablation inductively coupled plasma multi-collector mass spectrometer. The mass separation and ion detection unit is the MMS, as described in more detail above.

Possible sources of Error and Error Analysis

We have analyzed the primary, possible sources of error theoretically and they are elemental fractionation (unrepresentative sampling due to non-stoichiometric ablation; differences in mass transport, ionization efficiency, and ion extraction; and preferential loss of lighter species in differential pumping), heterogeneity of the mineral phases, and interferences.

In addition, one of the problems that can give rise to error in terrestrial labs is the formation of oxide species possibly by recombination in the cooler parts of the plasma. So, for example, $^{71}Ga^{16}O$ would give an interference at m/z 87 where Sr and Rb are measured. Our initial calculations show oxide interferences would be negligible for our instrument in use on Mars. For example, CeO is especially refractory and CeO/Ce, as a measure of oxide production, is typically ≤0.2% in well-tuned terrestrial Ar ICPMS plasma. We calculated the extent of interferences in a CO_2 plasma but in the absence of thermodynamic data for Ce, we used Sr as an analog and calculated the equilibrium composition of CO_2 with dilute quantities of Sr at elevated temperatures to estimate not only oxide but also carbide interferences. Interferences decrease with increase in plasma temperature but even at a modest 5000°K, SrO/Sr is 0.1% and SrC/Sr is even less.

In exactly the same way that there is mass interference between ^{40}Ar and ^{40}K, there is the same problem with ^{87}Sr and ^{87}Rb. Separating the ^{87}Rb peak from ^{87}Sr peak is very difficult and would require a mass spectrometer resolution of greater than 1 in 300,000. In terrestrial geochronology

labs this is solved by chemical separation of Rb and Sr using ion-exchange techniques. In this concept we will not separate the two elements but measure the spectrum which will include a combined Rb and Sr at m/z of 87. In the same way that it is possible to calculate the ^{40}K from the ^{39}K so the ^{85}Rb gives the ^{87}Rb and allows correction for its contribution to the combined Rb and Sr m/z 87. However, there are other, possible interferences caused by combinations of other elements that might be present in samples, but they too can be corrected for in a similar way.

We performed an error propagation analysis of the mass spectrum using concentrations of elements in Martian meteorite basalts [23, 24, 25, 26] (Figure 4) to assess achievable precision. We conservatively assumed 3% (2σ) relative precision on isotope values, our performance target for concentration analysis, using external standards. However, greater precision should be obtainable if we also use the Kr data to correct the ^{84}Sr peak and use ^{86}Sr/ ^{84}Sr as an internal standard with which other ratios are normalized. Using a 10,000 sample Monte Carlo simulation of measurements and corrections, the precision is 7% (2σ) for ^{87}Rb/^{87}Sr, as a proxy for a date, but an isochron date might have lower uncertainty. The error includes the uncertainty associated with analysis of materials for calibration.

This precision would make the dates applicable to many potentially accessible samples on Mars. Although this example of implementation has been focused on Mars, with the provision of an inert gas for the plasma the approach could be used for airless bodies or other targets.

Future Development

Our current efforts have made considerable progress in developing the concept of a low-mass, low-power radio frequency generator to produce the plasma. The next step would be to develop the interface between our plasma and the MMS and thus enable the first complete isotopic analyses with the system. At that stage, options for subtle revisions to the scheme would be evaluated and the design finalized.

4. Discussion

Relative Merits of the Various Dating Options

Although it may appear that our K-Ar approach is unique there is a possible alternative. In the most recent announcement of selections for the NASA Planetary Instrument Definition and Development Program (PIDDP) selections [27] one of the successful proposals was from Barbara Cohen of NASA Marshall Space Flight Center, to develop an instrument, *K-Ar Laser Experiment (KArLE) for In Situ Planetary Geochronology*. Unfortunately, few details are known.

In the case of rubidium-strontium dating, the two approaches use the same sampling technology but in all other respects could not be more dramatically different.

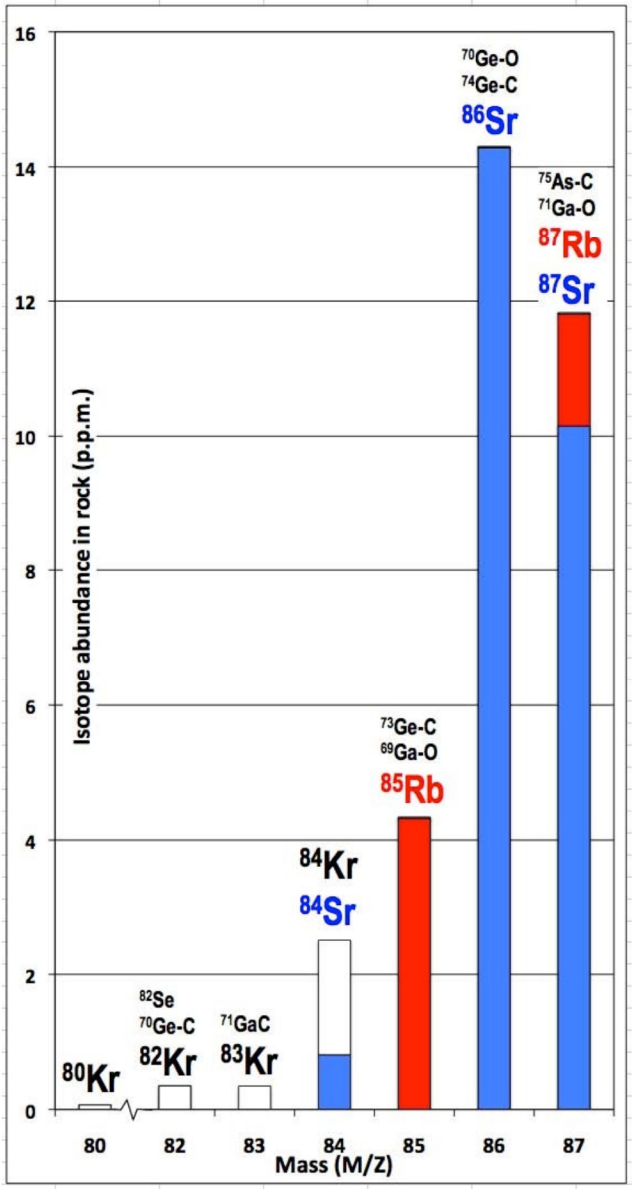

Figure 4. Modeled mass spectrum for Rb-Sr isotopic analysis, showing all isotopic species, that the interferences are negligible and how corrections would be made for mass interferences. The concentrations for interferences are from analyses of Martian meteorite basalt samples and Mars atmosphere. For example, correction for the contribution of ^{87}Rb to the ^{87}Sr peak is calculated by its ratio to ^{84}Rb, which in turn is corrected for the contribution of ^{84}Kr by reference to ^{80}Kr. All these relationships and consequent propagation of errors were included in the estimate of expected ^{87}Rb/^{87}Sr uncertainty of 7% (2σ).

Both have developed methods to overcome the problem of analyzing very small amounts of sample by, in one case using resonance ionization, and in our concept, an inductively coupled plasma, both to give nearly 100% ionization of a sample plume produced by the laser. It is clear that resonance ionization will give excellent separation of the different rubidium and strontium isotopes, however,

at the cost of deploying two very specialized lasers, both of which would need to be miniaturized. In our concept, we do not separate the rubidium and strontium isotopes but then suffer the increased error in correcting for that their mutual interference produces, but potentially with a saving in mass. In addition, our concept offers the opportunity of a broad range chemical analysis of any sample, which would give very valuable context information.

Mission Opportunities for Deployment of the Instruments

An initial evaluation of the current Mars explanation mission plans, to be executed jointly by NASA and ESA seems to give little hope for deployment of a geochronological instrument in the foreseeable future. The Max-C sample caching mission scheduled for 2018 launch is probably too soon for either concept to have achieved a sufficiently high TRL (Technology Readiness Level) to be selected. Also, the actual sample return mission to recover the cache is not planned in any detail both in terms of technology and an actual date at the moment. Furthermore, given the discrepancy between the histories of Mars exploration mission plans and their actual execution there may be scope for other major opportunities caused by delay or re-drafting of current plans.

Even with the current mission plan, the scientific pressure to get reliable ages of Martian material is very likely to increase well before any samples are returned to Earth. This will give encouragement for proposals to produce sufficiently reliable in situ geochronology measurements with dedicated lower-cost missions, which will require the availability of low-mass, low-power instruments to make the measurements.

However, Mars should not be thought of as the only target. There are many other solar system bodies with equally compelling problems to be attacked, once suitable technology has been developed.

5. CONCLUSIONS

We foresee several applications of either of the proposed instruments in enabling missions to Mars, small bodies or outer planets. Scientific objectives could include:

- calibration of the cratering time-scale (which also has implications for understanding cratering ages on other solar system bodies);
- dating of processes, e.g. formation of clays or evaporite minerals, such as sulfates);
- triage of outcrops to establish selections for sample return.

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7. ACKNOWLEDGMENT

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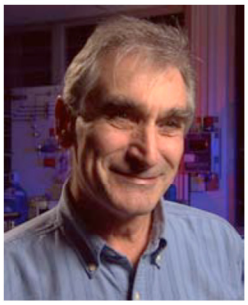
8. BIOGRAPHIES



Max Coleman is a Senior Research Scientist at NASA Jet Propulsion Laboratory, Caltech. His training combined chemistry, geology and isotope geochemistry at London and Leeds Universities, but he found that he needed to encompass microbiology too when studying sedimentary rocks. After ten years at the British Geological Survey, where he ran a national stable isotope facility, he joined BP to start a new geochemical research group. He then moved to the University of Reading in 1995 as Professor of Sedimentology. Max Coleman joined JPL in 2003, to be Director of the Center for Life Detection and Leader of the Astrobiology Research Group.

Michael Hecht, a Senior Research Scientist at Caltech's Jet Propulsion Laboratory, develops instruments for planetary science missions and conducts fundamental research on topics including martian polar processes, soil chemistry and microscopic structure, the physical chemistry of water in the soil and atmosphere, climate change, and polar paleoclimatology. He was Principal Investigator and Project Manager for the successful MECA instrument on the Phoenix Mars Scout mission, a soil analysis suite encompassing wet chemistry, microscopy, and thermophysical property investigations. Among his more recent technology development activities is a thermal drill designed for exploring the Polar Layered Deposits of Mars.

Joel Hurowitz is a Research Scientist at the Jet Propulsion Laboratory, working in the Planetary Science Section. His research interests deal with:

1. Understanding the origin and evolution of martian soils and sedimentary rocks by exploring the relevant fluid-rock reactions which control the alteration of primary igneous mineral phases and the formation of secondary minerals on Mars.

2. The development of novel measurement protocols that can be used to perform in-situ geochronology measurements on planetary surfaces.

Joel has also worked extensively on the Mars Exploration Rover mission as a member of the Athena science team.

Evan Neidholdt brings a background of experimental mass spectrometry to the team. Previous work includes development of novel devices and methods for ambient pressure ionization of explosives and CBW agents, for application with field portable mass spectrometry instrumentation. Currently, Evan is involved in system engineering of mass spectrometer instrument systems at JPL and current work includes delivery of a miniature JPL mass spectrometer to a stratospheric balloon platform for atmospheric analysis. He is a laboratory experiment lead for the JPL geochronology effort.

Jay Polk is a Principal Engineer in the Propulsion and Materials Engineering Section at the Jet Propulsion Laboratory, where he has worked for the last 21 years. Dr. Polk received a B.S. in Aerospace Engineering from the Georgia Institute of Technology and a Ph.D. in Mechanical and Aerospace Engineering from Princeton University. He is an expert in the area of high-current cathode physics, ion engine wear processes, high power plasma thrusters, and the application of probabilistic methods to analysis of engine service life. From 1997 to 2001 he served as supervisor of the Advanced Propulsion Group at JPL. He has authored over 100 papers in the field of electric propulsion and has received five awards for best paper at the International Electric Propulsion Conference and the Joint Propulsion Conference. Dr. Polk is also a Visiting Associate at the Graduate Aerospace Laboratories at Caltech where he teaches a graduate-level space propulsion class.

Mahadeva Sinha has been a member of technical staff at the Jet Propulsion Laboratory after obtaining a Ph.D. from Columbia University, New York in 1974. He possesses an extensive experience in miniature mass spectrometry, laser ablation and array detection of ions/electrons. Dr. Sinha is the inventor of the miniature mass spectrometer and array detector. He has also developed a new methodology based on the technologies of laser ablation-miniature mass spectrometer (LA-MMS) for chemical and isotopic analysis of rocks. Currently, he is working on the application of LA-MMS instrument for absolute age dating of rocks.

Wolfgang Sturhahn received his Ph.D. in physics in 1992 from the University of Hamburg, based on a thesis in nuclear resonant scattering with synchrotron radiation. He joined the Experimental Facilities Division of the Advanced Photon Source as visiting scientist in 1993 and accepted a staff position with Argonne National Laboratory in 1994. There he developed the nuclear resonant scattering beam line, discovered inelastic nuclear resonant scattering, a unique method for determination of vibrational density-of-states, and was responsible for the nuclear resonant scattering program. His research interests focus on high-pressure measurements for geophysical applications, vibrational studies on iron containing biomolecules and proteins, and the vibrational and magnetic behavior of thin films. In 2010 Sturhahn left Argonne as Senior Physicist and joined the Jet Propulsion Laboratory as senior-level Technologist. There he has been part of the JPL/Caltech in situ geochronology project, developed a high-vacuum Laser ablation system for static detection and isotope analysis of noble gases such as He, Ne, Ar, and provided quantitative data on the ^{40}Ar content of characterized mineral samples.

Wayne Zimmerman is currently the Chief Engineer for the Instruments and Science Data Systems Division at JPL. His current responsibilities include trouble shooting science instrument problems and insuring instruments meet performance, schedule, and cost requirements. He also is chairs both instrument design reviews and design teams for on-orbit and in-situ science instrument payloads for planetary, lunar, and Earth science missions. Wayne received his B.S. in Fluidics from Case Institute of Technology, Cleveland Ohio, and his M.S. in Aerospace Systems Engineering from the University of Southern California. Wayne has been at JPL for 34 years, designing and building robotic instrument delivery/sampling systems for Mars as well as designing integrated instrument platforms for terrestrial and planetary applications. He has received numerous NASA achievement awards as well as the NASA Exceptional Service Medal for engineering excellence.